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Indian Standard

METHOD FOR DETERMINATION OF SYNTHETIC PYRETHROIDS IN AGRICULTURAL AND FOOD COMMODITIES

ICS 65.100.01; 67.040; 71.040.50

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

April 2001

Price Group 1

Pesticide Residues Analysis Sectional Committee, FAD 34

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticide Residues Analysis Sectional Committee, had been approved by the Food and Agriculture Division Council.

Synthetic pyrethroids are characterized by a wide spectrum, high knock down and lethal action together with repellent and antifeedant activity. They have high ovicidal, larvicidal and adulticidal action on many insects. The synthetic pyrethroids applied over plants are absorbed by waxy layers where they remain to kill the insects. They are rapidly detoxified by the soil microorganisms. The most commonly used synthetic pyrethroids are permethrin, cypermethrin, fevalerate and deltamethrin. This standard will enable the regulatory authorities and workers in this field to follow an uniform test procedure for quantitative determination of these pesticides.

In the preparation of this standard, due consideration has been given to the limits laid down under the *Prevention of Food Adulteration Rules,* 1954 and rules framed thereunder. This standard will enable the health authorities and other engaged in the field to follow uniform test procedure for the estimation of residues in environmental samples and food commodities.

In reporting the results of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHOD FOR DETERMINATION OF SYNTHETIC PYRETHROIDS IN AGRICULTURAL AND FOOD COMMODITIES

1 SCOPE

This standard prescribes a gas chromatographic method of determination of synthetic pyrethroid residues viz. permethrin, cypermethrin, deltamethrin and fenvalerate, in agricultural and food commodities.

2 REFERENCE

The following standard contains provisions which through reference in this text, constitutes provisions of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No.	*Title*
11380 : 1985	Methods of sampling for the determination of pesticide residues

3 SAMPLING

Representative samples for the purpose of estimation of synthetic pyrethroids shall be drawn in accordance with IS 11380. Samples should be stored at 20°C till they are processed.

4 APPARATUS

4.1 Gas Liquid Chromatograph (GLC)

GLC shall be fitted with an electron capture detector (Ni^{63}) and an integrating-cum-recording device. The following operating parameters are suggested:

Column : Stainless steel, 2 m × 1/8 inch i.d. packed with 3 percent OV — 10-1 on 80/100 mesh Chromosorb W-HP.

Temperature :

Column oven	:	260°C
Injector	:	280°C
Detector	:	300°C
Carrier gas flow	:	30 ml/min

4.2 High Speed Blender

4.3 Rotary Vacuum Evaporator

4.4 Separatory Funnels — 500 ml capacity.

4.5 Buchner Funnel and Filtering Flask

4.6 Crolitre Syringe of 10 µl Capacity

4 REAGENTS

4.1 n-hexane — Extra pure or equivalent grade.

4.2 Acetone — AR grade.

4.3 Anhydrous Sodium Sulphate

4.4 Aluminium Oxide (Neutral) — column chromatography grade

4.5 Florisil (60-100 mesh)

4.6 Reference Standards of Permethrin, Cypermethrin, Fenvalerate and Deltamethrin

4.7 Activated Floronil 9.8-2.1 (NaCl) Sodium Chloride Solution

5 SAMPLE PREPARATION

5.1 Cereals, Pulses, Condiments, Tea, Coffee, Sugar, etc

If needed grind to pass through 250 micron IS sieve draw 100 g by mixing.

5.2 Vegetables and Fruits

5.2.1 Remove all visible soil particles by gentle rubbing. Scrub root vegetables with a brush and wash.

5.2.2 Remove all inedible portions of vegetables, such as, outer leaves of cabbages, carrot tops, stems of tomatoes, stones of fruits, etc. Remove the peel of the skin of a fruit if it is normally not consumed, such as, peas, oranges, etc. Do not remove the skin if it is edible, such as apples, carrots, etc.

5.2.3 Cut a representative sample into pieces and reduce to about 100 g by mixing and quartering.

5.3 Oilseeds and Nuts

Remove an discard shells if present. Reduce the sample to the desired size for extraction by mixing and quartering. Chop the sample finely.

5.4 Hydrogenated and Unhydrogenated Vegetable Oils

These do not require any preparation.

5.5 Water and Beverages

These do not require any preparation.

6 EXTRACTION

Blend 25 g of the sample in a glass jar with 100 ml solvent mixture (a:1 hexane: acetone *v/v*) and 25 g sodium sulphate at high speed for 1.5 min. Filter the homogenized material and re-extract twice with additional 70 ml aliquots of solvent mixture, pool the filtered extracts.

7 PARTITIONING

Transfer the filtered extract to a 500 ml separatory funnel, dilute with 200 ml of 2 percent sodium sulphate solution and shake for 2 min. Allow the phases to separate. Discard the acetone layer and drain the hexane layer through 2 cm layer of anhydrous sodium sulphate to collect into a 250 ml round bottom boiling flask. The rinsings of separatory funnel with 10 ml hexane should be treated similarly. Concentrate the combined hexane extract under reduced pressure at 40°C to approximately 5 ml in rotary vacuum evaporator.

8 CLEANUP

Wash a glass column packed with 10 g grade III neutral alumina and 5 g florisil overlaid with 2 cm layer of anhydrous sodium sulphate slurry with 50 ml hexane. Transfer the above hexane extract quantitatively through the above column. Elute the cleaned up residues from the column with 200 ml of 9 : 1, hexane : acetone *v/v* solvent mixture. Dry this eluate in a rotary vacuum evaporator under reduced pressure and dissolve the residues in 5 n-hexane.

9 ESTIMATIOIN

Inject 3 µl of the standard solution or the above cleaned up extract into the GLC. Identify the peaks by their retention times and record the peak areas. Under the GLC conditions listed above the retention times of the pyrethroids were as follows, permethrin — 1.7 min, cypermethrin — 2.3 min, fenvalerate — 2.9 min, deltamethrin — 3.5 min.

10 CALCULATION

$$\text{Residues in ppm} = \frac{A_s \times V_{std} \times C_{std} \times D_f \times F}{A_{std} \times V_s \times W_s}$$

where

A_s = peak area of sample injected,

A_{std} = peak area of standard injected,

V_s = volume of sample injected,

V_{std} = volume of standard injected,

C_{std} = concentration of standard,

W_s = weight of sample taken in gm,

D_f = dilution factor, and

F = recovery factor
= 100 percent mean recovery.

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